

In re Application of:	:	
<i>Bruce J. Kokko</i>	:	Examiner: S. Alvo
U.S. Serial No. 09/456,270	:	Group Art Unit: 1731
Filed: December 7, 1999	:	
Docket No. 2130 (FJ-99-12)	:	
For: METHOD OF MAKING ABSORBENT SHEET FROM RECYCLE FURNISH	:	

DECLARATION OF BRUCE J. KOKKO

1. That he was awarded a Ph.D. degree in Chemistry from the University of Illinois, Urbana, Illinois, in 1983. Since that time he has worked on projects involving chemistry relevant to the manufacture of paper, including sheet useful for making paper towel and paper tissue.
2. That he is familiar with the above-noted application, the Official Action of February 15, 2002, as well as the following references:

Osborn, III, United States Patent No. 4,351,699

3. That when recycle furnish is used in the manufacture of sheet for use in paper towel and paper tissue, the sheet is frequently undesirably stiff and has undesirably low softness. These characteristics correlate with high tensile strength and the problem is most severe when 100% recycle fiber is used. In order to reduce stiffness and promote softness, debonder is typically used which also has the effect of reducing tensiles. The "softness" increase correlates with reduction in tensile strength.
4. During the course of his research, he unexpectedly found that: (a) quaternary ammonium surfactants and nonionic surfactants can exhibit large synergism with respect to their debonding characteristics; (b) the synergy correlates with the hydrophile-lipophile (HLB) value of the nonionic surfactant and depends on the organic (hydrophobic) chain length present in the nonionic surfactant; and (c) the synergy is particularly useful when large tensile reductions are sought as would be the case when 100% recycle fiber is used when making a sheet.
5. That the process of the invention employing synergistic surfactant combinations was first postulated in connection with the experiments summarized in Table 1 of the above-noted application, reproduced below:

Table 1: Tensile reduction data for debonder/softener formulations.

Formulation	Quat 1 <sup>A</sup> (mol/ton)	Tensile Reduction (%)	Quat 2 <sup>B</sup> (mol/ton)	Tensile Reduction (%)	Additive Tens. Red. (%)	Observed Tens. Red. (%)
H	0.73	7	1.45	4	11	20
	1.95	18	3.86	6	24	19
J	1.11 <sup>C</sup>	7	1.68	4	11	14
	2.97 <sup>D</sup>	18	4.47	6	24	26
B <sup>D</sup>	0.19	2	0.37	2	4	20
	0.51	5	0.98	3	8	32

- A) Dimethylditallowammonium chloride  
 B) Di-(2-hydroxyethyl) - methylstearyl ammonium chloride  
 C) Behenyl-trimethyl ammonium chloride instead of Quat 1  
 D) Formulated with 33 wt.% PEG-6-dilaurate

The third "row" (really 2 rows) of Table 1 includes a summary of observations made with the debonder compositions of Example series B, C and F of the above-

noted application as is also summarized in Figure 2 of the application. The third, fifth and sixth columns set forth individual and expected additive tensile reductions based on the quaternary surfactants of Examples C and F of the patent application. The last or seventh column of Table 1, row 3 is the observed tensile reductions seen with the debonder composition of Example series B. The observed tensile reductions with the composition of Examples series B were unexpectedly high at 4-5 times the additive tensile reductions of Example series C surfactant and Example series F surfactant, which include the individual quaternary compounds of the debonder used in Example series B. The debonder used in Example series B additionally contained PEG-6-dilaurate, a C12 nonionic surfactant with an HLB number of less than 10.

6. That based on the above observations, he postulated that enhanced tensile reductions in recycle fiber products could be achieved by combining a nonionic surfactant with an imidazolinium quaternary salt surfactant and formulated Example I, which is a novel composition which outperformed the debonder composition of Example series B. *See* Figure 2 of the above-noted application. The hypothesis was correct as can be seen especially in Examples O and P of the above-noted application.
7. That upon further investigation, he discovered that the debonding capability of a quaternary surfactant/nonionic surfactant combination depended upon the HLB number of the nonionic surfactant employed. This discovery was also unexpected, and is summarized in Table 3 of the application, set forth below:

Table 3: **HLB** for Maximum Debonding

No. Carbon Atoms in Fatty Acid	Preferred <b>HLB</b> for PEG-Mono R	Preferred <b>HLB</b> for PEG-di-R
R = C12	>10	<10
R = C18	>10	>10

8. That especially preferred nonionic surfactants for use in connection with the process of the present invention include the following due to their ability to reduce tensile:

(a) monoalkylated nonionic surfactants such as alkoxyated fatty acids or alkoxyated fatty alcohols having an **HLB** value of greater than about 10 wherein the fatty acids and fatty alcohols have 12 carbon atoms or more; (b) dialkylated nonionic surfactants such as alkoxyated fatty acids or alkoxyated fatty alcohols with an **HLB** value of greater than about 10 wherein the fatty acids or fatty alcohols have about 16 carbon atoms or more.

9. The known debonder composition used in Example series A of the above-noted application included PEG-6-dioleate as a nonionic surfactant which has a C18 fatty acid (lipophillic) carbon chain length and an HLB value of less than 10 and thus does not meet the nonionic surfactant HLB value criteria set forth in any of the amended claims. Also, the debonder composition of Example A contained less than 25% by weight nonionic surfactant which is also recited in the claims, as amended, in the above-noted patent application.

10. That, in his opinion, the unexpected results enumerated above are not suggested in any way by United States Patent Nos. 5,730,839; 5,582,681 or 4,351,699 cited by the Examiner because these patents fail to disclose that combinations of quaternary surfactants and nonionic surfactants can exhibit large synergies and the synergies depend on the HLB value of the nonionic surfactant.

11. The undersigned Declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States

Code and that such willful false statements may jeopardize the validity of the subject application or any patent issuing thereon.

Dated

6-12-02



Bruce J. Kokko